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TECHNICAL REPORT
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LOW-TEMPERATURE TRACTION RUBBER SOLE
COMPOUND FOR DMS COMBAT FOOTWEAR

by

Vincent S. Javier

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NATICK LABORATORIES
Natick, Massachusetts 01760



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Clothing and Organic Materials Division

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TECHNICAL REPORT

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LOW-TEMPERATURE TRACTION RUBBER SOLE COMPOUND
FOR DMS COMBAT FOOTWEAR

by

Vincent S. Javier
Rubber, Plastics and Leather Engineering Branch

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Clothing and Organic Materials Division
U. S. ARMY NATICK LABORATORIES
Natick, Massachusetts 01760

FOREWORD

There is a need for a new rubber sole and heel compound for the Army combat footwear readily produced by application of the direct molded sole process. Although the presently adopted standard sole compound has been exhibiting satisfactory wear properties and durability, it does not provide the combat boot with adequate traction at low temperature and on snow and ice. This deficiency of the present outsole compound was confirmed by the U. S. Army troops who conducted an actual wear test in Alaska on the newly adopted combat boots.

It was for this reason that this laboratory initiated a rubber compounding development program aimed at obtaining a soling material which would provide both adequate traction on snow and ice and satisfactory wear serviceability.

This report discusses the development and evaluation of the new rubber compound designed solely for the Army combat boot with the direct molded sole construction.

S. J. KENNEDY
Director
Clothing and Organic Materials Division

APPROVED:

DALE H. SIELING, Ph.D.
Scientific Director

W. M. MANTZ
Brigadier General, USA
Commanding

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ABSTRACT

A new direct molded sole rubber compound designed primarily to give better traction at low temperature, and on snow and ice, than the present standard vinyl-modified Buna N rubber compound, has been successfully developed by utilizing Styrene-Butadiene rubber (SBR), type 1502.

The vulcanizates of this new compound exhibited good physical properties which are comparable with the physical properties exhibited by the present standard sole compound. In addition, this new compound also displayed excellent ozone resistance and good low-temperature behavior - a property which makes this compound more suitable for wear on snow and ice.

Furthermore, the test results on the finished boot samples showed that the new SBR compound has satisfied not only the curing conditions imposed by the CEMA and Welco process but also the physical requirements, except for solvent resistance, which are specified in Military Specification, MIL-B-43154, for the standard direct molded sole (DMS) combat boot.

IMPROVED LOW-TEMPERATURE TRACTION RUBBER SOLING FOR DMS FOOTWEAR

I. Introduction

One of the many items developed by the U.S. Army Natick Laboratories under the Army Research and Development program is the Army combat boot with the direct molded sole construction. The development of this item was accomplished by utilizing the technique brought about by the CEMA process⁽¹⁾. This technique fabricates shoes by molding and bonding in one operation the sole and heel to the bottom part of the leather shoe upper. The new technique, therefore, has eliminated stitching, nailing, and cementing, which were the conventional operations used in attaching rubber soles and heels to the boot uppers. Another good feature of this process is an improvement in the end product; that is, the rubber bond is not only made more waterproof than the conventional construction, but contributes to much longer shoe life.

In contributing to the successful development of the direct molded sole combat footwear, the Rubber Technology Group of the Clothing and Organic Materials Division undertook intensive compounding development of rubber stocks that could perform satisfactorily under the curing conditions and problems imposed by the CEMA Vulcanizing Process.

The process of vulcanizing the sole and heel directly to the shoe upper consists of inserting a lightweight aluminum metal last into the shoe upper to provide some degree of rigidity. The lasted shoe upper, firmly held in locked position, is then placed over the sole mold.

The mold consists of three parts, a bottom plate and two side plates. The two side plates are cut to the proper contour and size to form the edges of the sole. Before placing the lasted upper over the sole mold, a weighed amount of unvulcanized rubber sole and heel is placed on the bottom plate and the curing cycle is started automatically. The side plates move in against the lower edge of the shoe to form a tight seal and then the bottom plate moves upward, forcing the uncured compound against the shoe upper bottom and out into the mold.

The force for closing the side and bottom plates is provided by hydraulic pressure acting on the pistons attached to the plates. The heat for vulcanization of the sole and heel compound is supplied by electric heaters inside the bottom and side plates of the mold.

At the end of the curing cycle, the pressure is released and the side molds open to the full extent of their travel. The sole mold is withdrawn and the shoe is left free on the last away from the source of heat. The final step is removal of the metal last from the finished shoe.

Basically, this technique of vulcanizing the sole and heel to the leather upper has presented problems in the formulation of a satisfactory sole and heel rubber compound. One of the problems is the low molding pressure. The side mold pressure must be kept low to prevent cutting and other damages to the leather of the shoe by the side plates. The pressure on the bottom plates should also be low to prevent the compound from flowing through the seal formed by the side plates and the shoe. Thus, the CEMA process maintains operating pressure much lower than the operating pressure used by the conventional rubber vulcanizing process.

One other problem to consider with this process is the low heat input for vulcanization. Since the leather from which the shoe is made is not resistant to high temperature, the side plates' temperature must be kept as low as practicable to prevent deterioration of the leather during vulcanization. Furthermore, the major heat for vulcanization is applied only to the bottom plates of the sole mold and to a small area of the side mold. And since there is no direct heat contact between the bottom plates and the surface upon which the sole is molded, the problem of obtaining a satisfactory cure which would give the molded-on-sole satisfactory wear and adhesion properties at the interface of the rubber and the leather becomes even more difficult.

The short vulcanization cycles pose another problem. Normally, the direct molded sole process is designed to produce one pair for each cycle. This, again, is in contrast to the conventional procedure of using multicavity molds and multi-platens process. Thus, for efficiency and economy, the curing cycle must be as short as possible to enable a maximum hourly output of finished shoes per vulcanizer.

To perform satisfactorily under the set of curing restrictions cited above, the compound should be fast curing and should have an excellent flow under low pressure. For example, a representative compound might be satisfactory with respect to the rate of cure but not acceptable for adequate mold flow. On the other hand, the mold flow might be satisfactory but the cure rate might be too slow and the compound would not be acceptable.

II. Development of Polyblend Buna N Rubber Compound for Direct Molded Sole Footwear

The development of a polyblend rubber compound started when this new type of elastomeric compound was first introduced commercially. Since this was a new product, this laboratory undertook evaluation studies of it for military applications. It was found that the vulcanizates of this new material exhibited amazingly good physical properties, including excellent gasoline and ozone resistance. At that time, gasoline resistant soling for combat footwear was being considered, and, for this reason, the program of developing this new polyblend nitrile rubber for direct

molded sole combat footwear was initiated. A series of compounding formulations were tried and, finally, under the joint effort of Naugatuck Chemical Division of U.S. Rubber Company and this laboratory, a sole and heel formulation which performed satisfactorily under both the curing and molding conditions of CEMA process was obtained.

Using this formulation, Endicott Johnson Corporation, under contract, fabricated the first samples of direct molded sole combat footwear which were then subjected to actual wear test by Army troops. Wear test results, however, indicated failure of the boots to maintain satisfactory wear life. Sole and heel separations from the shoe upper, due to weak bond, were found to be the most consistent failures. This type of failure was quickly overcome by developing an improved vulcanizing rubber cement for use with this compound for direct molded sole process. This improved cement has now given the Army direct molded sole footwear excellent wear durability. The rubber compounding recipe is given in Table XI and test results are shown in Table XII.

III. Development Compounding of Styrene-Butadiene for DMS Footwear

The reported unsatisfactory traction performance on snow and ice⁽²⁾ of the direct molded sole combat leather boot with the standard vinyl-modified Buna N rubber compound created the need for a new rubber soling material which would give improved traction qualities at low temperature and on snow and ice.

An attempt, therefore, was made by this laboratory to develop a soling compound which would provide combat footwear with adequate traction, particularly on snow and ice. Development effort was directed toward obtaining softer compounds which will remain flexible and soft in temperatures as low as 0°F, thus allowing greater retention of traction characteristics by the outsole at lower temperatures⁽³⁾.

Exploratory compounding was then undertaken, and a series of formulations utilizing most of the available general purpose rubbers, such as Natural, Polychloroprene, Chlorosulfonated Polyethylene, Styrene-Butadiene, and even blends of cis-Polybutadiene with vinyl-modified Buna N and with other rubbers, were explored. The compounding recipes and properties of these compounds are given in Tables I to V.

Compounds which displayed desirable properties were carefully noted. Among those compounds worth investigating was the Polychloroprene-containing compound. Two candidate compounds were evaluated and DMS combat footwear samples, using compound 63-C-45, were fabricated by this laboratory and were actually wear tested⁽⁴⁾ by some Army troops at Fort Lee, Virginia, proving ground. Table VI gives the recipes used in development compounding and the properties of the compounds. Difficulties in mass producing this compound led to continued development for a new compound.

The polyblend-containing compounds, likewise displayed excellent abrasion resistance and these compounds also were worth investigating because blending cis-Polybutadiene with vinyl-modified Buna N, which is the rubber used in obtaining the present DMS compound, gave significant improvement to abrasion characteristics. The recipes used in compounding and the properties obtained are given in Table VII.

Although the Polychloroprene and polyblend-containing compound exhibited desirable properties, neither one of these compounds was considered to satisfy the Army's need for a new DMS rubber compound with improved traction on snow and ice. The personnel responsible for undertaking this project believed that a cheaper and a better quality sole compound could be obtained with Styrene-Butadiene-containing compound. The good low-temperature behavior, easy processing characteristics and cheaper cost of the Styrene-Butadiene rubber are the chief reasons for selecting Styrene-Butadiene-containing compound for the development of the needed sole and heel material.

Three phases of compounding development work were, therefore, performed on the SBR-containing rubber. The first was to obtain an ozone-resistant compound. SBR is known to be susceptible to ozone attack, but its compatibility to blend with Ethylene-Propylene Diene rubber (EPDM)⁽⁶⁾ made it possible to obtain an SBR compound with excellent ozone resistance. Table VIII gives the recipes used in compounding and the test results obtained.

The next phase was compounding for abrasion resistance. SBR compound reinforced with hydrated silica does not usually display high abrasion performance. However, good physical property, including abrasion resistance, could be obtained with SBR 1502, reinforced with HiSil 233, by the omission of zinc oxide⁽⁵⁾ in the compound. Normally, zinc oxide is needed in conventional rubber compounding as an activator and stabilizer. Table IX gives the effect of varying the zinc oxide loading in the compound and the recipes used.

Lastly, the compounding development included obtaining a stock which would perform satisfactorily under the Military Specification requirement for sole and heel compound and under the curing and molding conditions of the CEMA vulcanizing process. The Mooney viscometer was then employed to measure the cure rate and flow characteristics of the candidate compounds. The recipes used in compounding and the properties obtained are shown in Table X. The best overall properties are exhibited by compound SBR-29b and, therefore, represented the new rubber sole compound for the DMS combat footwear. The new compound, whose formulation is given in Table XI, was evaluated against the present DMS compound, and Table XII gives the comparative properties of the two compounds.

IV. Experimental and Test Procedures

All rubber compounding and vulcanization were done in this Laboratory with the conventional standard laboratory rubber-processing equipment.

In addition to the prepared and molded-slab samples for evaluation, samples of combat boots were also fabricated, utilizing the CEMA equipment available in these Laboratories, to determine the molding and curing characteristics of the compound when processed by the DMS technique.

The vulcanizing rubber cement used in molding the rubber sole and heel directly to the leather upper was also prepared according to the recipes listed in Table XIII, Vulcanizing Cement for Vinyl-Modified Buna N Standard Compound and Table XIV, Vulcanizing Cement for SBR Compound.

The new SBR compound was further evaluated in production quantities by Wellco Ro-Search, Inc. One hundred pairs of DMS boots were made by this Company for evaluation. Wellco Ro-Search production personnel, however, believed that the new soling compound needed modification to suit their processing and molding equipment. A slight modification of the compound was made, and, using this modified formula, the one hundred pairs of combat boots were fabricated. The modified formulation for both rubber and cement compound is given in Table XV.

Laboratory tests were also performed on the Wellco Ro-Search fabricated boots and the results are tabulated in Table XVI. The boots are presently under wear test.

The following test methods were used in the evaluation of these properties:

<u>Property Tested</u>	<u>Fed. Test Method, Std 601</u>
Tensile strength	4111
Ultimate elongation	4121
Stress @ 300% elongation	4131
Hardness	3021
Abrasion	14111
Volume swell	6211
Cure characteristics	ASTM Designation D1646
Cutgrowth	ASTM Designation D1052
Ozone resistance	Test was run according to Par. 4.4.5, Test for Ozone Resistance, of MIL-B-43154
Bond strength	Test was run according to Par. 4.4.6 of MIL-B-43154

Bond strength by dead weight application was run in accordance with Par. 4.4.6 of MIL-B-43154 except that a 20-pound dead weight was used for separation instead of the machine method of ASTM D-413.

V. Results and Discussion

It was shown in Table VI, Development of Polychloroprene-Containing Compounds, that Neoprene WRT can be compounded to give a good quality non-mark sole and heel compound.

Likewise, test results obtained with Polyblend-containing compounds listed in Table VII showed significant improvement in abrasion resistance of vinyl-modified Buna N when blended with cis-Polybutadiene-type rubber in varying amounts.

With the development compounding of Styrene-Butadiene-containing compounds, it was shown by test results in Table VIII that SBR 1502 can be definitely protected from ozone attack by blending it with Royalene X-400, an oil-extended Ethylene-Propylene Diene rubber, plus addition of antiozonant in normal amount. Likewise, the test results obtained with compounds listed in Table IX, Effect of ZnO Loading in SBR 1502, have shown that the most desirable properties of SBR 1502, reinforced with HiSil 233, can be obtained by omitting zinc oxide, which is normally needed in conventional rubber compounding.

Finally, the new compound SBR-29b, listed in Table XI, was evaluated against the present standard DMS compound. The test results, as shown in Table XII, showed the experimental compound to have properties, excepting gasoline resistance, comparable with the properties of the standard sole compound. This would indicate that satisfactory wear performance can be expected of this newly developed compound. The hardness property at 0°F, on the other hand, indicated that the SBR compound displayed less tendency to harden than the vinyl-modified Buna N compound. Again, this would demonstrate that the new SBR soling compound can be expected to provide better traction at low temperature and on snow and ice.

In addition, the test data obtained with the Wellco Ro-Search modified SBR compound (listed in Table XV with test results on both the molded slab samples and finished boots shown in Table XVI) also compared favorably with the test data obtained with the original experimental compound SBR-29b. Furthermore, the test data also showed that the new compound can be satisfactorily produced by the direct molded sole technique.

VI. Conclusions

- a. A high quality non-mark soling compound with very desirable

physical properties, excellent ozone resistance and good low temperature serviceability for direct molded sole combat footwear can be obtained with Styrene-Butadiene rubber type 1502.

b. A soling material with an unusually high abrasion characteristic and with good gasoline resistance can be obtained by blending cis-Polybutadiene rubber with vinyl-modified Buna N.

VII. Recommendation

It is recommended that the new SBR base rubber compound be adopted for use in the Army Boot, Combat, Man's, Leather, Black, Direct Molded Sole, Mildew Resistant.

VIII. Acknowledgments

The author wishes to express his appreciation for the advice offered him by Mr. C. B. Griffis, for the assistance of Mr. Leonard Lulka with the milling, mixing, and curing operations and Mr. Charles Shurtleff with the physical testing, and Mr. D. S. Swain and Mr. L. H. Hollier for assistance in obtaining production quantities of boots.

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2. U.S. Army Test and Evaluation Command, U.S. Army Infantry Board, Fort Benning, Georgia, Project No. 8-4-6001-01, Final Report of Check Test of Boot, Combat, Leather, Direct Molded Sole, August 1965.
3. Elastomer Branch Report No. 41, "Investigation of Factors Affecting the Frictional Characteristics of Rubber Soling Material," by Angus Wilson, U.S. Army Natick Laboratories, Natick, Mass, September 1961.
4. U.S. Army Quartermaster Research and Engineering Field Evaluation Agency, Fort Lee, Virginia, Final Letter Report, Engineering Design Test of Boots, Combat, Leather, DMS Conventional vs. Special Soling, TECOM 8-3-6000-02K, 26 May 1964.
5. Pittsburgh Plate Glass Company, Pittsburgh, Pa., Technical Service Bulletin, "HiSil in SBR Compounds," 6 October 1964.
6. Clothing and Organic Materials Division Technical Report No. 7, "Silica-Reinforced Ethylene-Propylene Diene Rubbers," by Peter Dunn and Vincent S. Javier, U.S. Army Natick Laboratories, Natick, Mass, August 1965.

TABLE I. NATURAL RUBBER-CONTAINING COMPOUNDS: EXPLORATORY COMPOUNDING
(Recipes and Properties)

Materials*	63-N-1	63-N-2	63-N-3	63-N-8	63-NB-1	63-NB-2	63-NB-3	63-NB-8
a. Recipes								
Natural rubber	100	100	100	100	60	80	80	70
Budene 501					40	20	20	30
Zinc oxide	5	5	5	5	5	5	5	5
Stearic acid	1	1	1	1	1	1	1	1
HiSil 233	55	55	50	55	55	55	50	55
NPC Black	3	3	3	3	3	3	3	3
Agerite powder			2	2				2
Neozone D	2	2			2	2		
UOP 88	2	2			2	2		
Flexzone 3C			2	1			2	1
Cumar P25	3	2	3		3	3	3	
Triethanolamine	1.5	1.5	1.5	2	1.5	1.5	1	2
Reogen	2	2			2	2		
Light process oil	10	10	10	15	10	10	10	15
Captax	1.2	1.2			1.2	1.2		
Thionex	0.5	0.5			0.5	0.5		
DOTC			0.75	0.75			0.75	0.75
Santocure			1.5	1.5			1.5	1.5
Sulfur	2.5	2.5	2.5		2.5	2.5	2.5	
TP-90-B			5	2			5	2
Flexol TOF	5	5			5	5		
b. Properties at (10 or 12 min cure time/310°F) as indicated								
	(10/310)	(10/310)	(10/310)	(12/310)	(10/310)	(10/310)	(10/310)	(12/310)
Hardness, Shore A.-Orig.	80		70	70	79		70	68
After 1 hr. at 0°F	87	40	78	78	84	100	76	76
Abrasion index, -Orig.	58	40	87	67	124	100	94	91
After 70 hr @ 212°F		40		57		100		75
Cutgrowth, Orig.	0	0	0	0	0	0	0	0
After 70 hr @ 212°F		0	0					150

* See Appendix A for identification of materials listed by trade name.

TABLE II. STYRENE-BUTADIENE-CONTAINING COMPOUNDS: EXPLORATORY COMPOUNDING
(Recipes and Properties)

Materials*	63-SB-1	63-SB-2	63-SB-3	63-SB-4	63-SB-5	63-SB-6	63-SB-7	63-SB-8	63-SB-9	63-SB-10
	a. Recipe									
SBR 1500	60	50				50	50	50		
SBR 1502			50						50	50
SBR 1504				50						
SBR 1023	20				50					
Budene 501	20	50	50	50	50	50	50	50	50	50
SBR 1712c										
HISil 233	40							50	50	50
Phil Black 0	3	3	3	3	3	3	3	3	3	3
Mistron vapor		75	75	75	75					
Paragon clay										
Agerite Resin D	1	1	1	1	1					
Octamine						1	1	1	1	1
Flexzone 3C							1.5	1.5	1.5	1.5
UOP 88	2	1.5	1.5	1.5	1.5	1.5	1.5	1.5		
UOP 688										
Sunproof Jr.	1									
Sunolite wax		1	1	1	1	1	1	1	1	1
NALCO L-1718		3	3	3	3					
ZnO		5	5	5	5	5	5	5	5	5
Light process oil										
Flexol TOF	10	10	10	10	10	10	10	10	10	10
Diethylene glycol	2					1.5	1.5	1.5	1.5	1.5
Stearic acid	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Dutrex 20						5	5	5	5	5
Altax	1	2	2	2	2	2	2	2	2	2
Captax	1.5									
DOTG										
DPC						1	1	1	1	1
Methyl tuads	0.5	0.25	0.25	0.25	0.25					
Sulfur	2.5	2	2	2	2	2.5	2.5	2.5	2.5	2.5

* See Appendix A for identification of materials listed by trade name.

TABLE II (Cont'd) STYRENE-BUTADIENE-CONTAINING COMPOUNDS: EXPLORATORY COMPOUNDING

63-SB-1 63-SB-2 63-SB-3 63-SB-4 63-SB-5 63-SB-6 63-SB-7 63-SB-8 63-SB-9 63-SB-10

b. Properties at 10 min cure time/300°F

Hardness, Shore A									
Original	62	59	59	55	55	67	67	65	66
After 1 hr @ 0°F	70	63	62	60	58	70	70	68	70
Abrasion Index									
Original	70	25	32	23	20	123	113	200	166
After 70 hr @ 212°F									
Cutgrowth after									
50,000 flexes									
Original	500	500	500	500	500	500	500	100	200
After 70 hr @ 212°F									

a. Recipes (Cont'd)

Materials*	63-SB-11	63-SB-12	63-SB-13	63-SB-14	63-SB-15	63-SB-16	63-SB-17	63-SB-18	63-SB-19
SBR 1504	50		50		50	50	50	50	
SBR 1712c		50		50					
Budene 501	50	50	50	50	50	50	50	50	50
SBR 1778									
Agerite powder	1	1	1	1					
Octamine	1	1	1	1	1	1	1	1	
Flexzone 3C	2	2	2	2					
ZnO	5	5	5	5	5	5	5	5	5
Stearic acid	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1
HS11 233	40	40	35	35	40	40	40	40	55
Mistron vapor	15	15	20	20	15	15			
Phil Black 0	3	3	3	3	3	3	3	3	3
Paragon clay									
Light process oil	10	10	10	10	10	10	10	10	15
Dutrex 20	5	5	5	5	5	5	5	5	

* See Appendix A for identification of materials listed by trade name.

TABLE II (Cont'd) STYRENE-BUTADIENE-CONTAINING COMPOUNDS: EXPLORATORY COMPOUNDING

Materials*	63-SB-11	63-SB-12	63-SB-13	63-SB-14	63-SB-15	63-SB-16	63-SB-17	63-SB-18	63-SB-19
a. Recipes (Cont'd)									
Sunproof Jr.	1	1	1	1	1	1	1	1	1
UOP 88					2		2		
UOP 688						2		2	
Diethylene glycol	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Altax	1	1	1	1	1	1	1	1	2
DRG									1
Captax	1	1	1	1	1	1	1	1	
Methyl tuads	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	
Sulfur	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	
b. Properties at 10 min cure time/310°F (Cont'd)									
Hardness, Shore A									
Original	69	66	68	65	67	67	66	67	68
After 1 hr @ 0°F	78	77	77	76	75	75	73	72	72
Abrasion Index									
Original	99	78	79	68	95	96	108	97	76
After 70 hr @ 212°F									
Cutgrowth, after									
50,000 flexes									
Original	300	500	300	500	250	350	300	500	300
After 70 hr @ 212°F									

* See Appendix A for identification of materials listed by trade name.

TABLE III. CHLOROSULFONATED POLYETHYLENE-CONTAINING COMPOUNDS: EXPLORATORY COMPOUNDING
(Recipes and Properties)

Materials*	63-CS-7	63-CS-MA-7	63-CS-8	63-CS-9	63-CS-10	63-CS-11	63-CS-B-1	63-CS-B-2	63-CS-B-3	63-CS-B-4
	100	90	100	100	100	100	80	80	70	80
Hypalon 40										
Hycar 1072		10								
Budene 501							20	20	15	20
SBR-1023										
NBC	2	2	2	2	2	2	2	2		2
UOP 88	1	1	1	1	1	1	1	1	1	
HiSil 233	25	25	25	25	25	25	25	25	35	
SRF Black	3	3	3	3	3	3	5	5	5	5
Sunolite wax	1	1	1	1	1	1	1	1	1	1
XLC Magnesia	5	5	5	5	5	5	5	5	5	5
Butyl oleate	10	10	5	20	20	5	10	10	5	5
TP-90-B	20	20	15	20	5	15	10	10	5	15
Butyl stearate	5	5	5	5	5	5	5	5		
Litharge	5	5	5	5	5	5	5	5	5	5
DIDA									20	
Flexzone 3C						2				2
Mistron vapor						45				45
Thermoflex A						1				1
Tetrone A	2	2	2	2	2	2	2	2	2	2
Altax	1	1	1	1	1	1	1	1	1	1
Sulfur										
b. Properties at indicated (cure time, min/temp °F)										
Hardness, Shore A	(10/310)	(10/310)	(10/310)	(10/310)	(10/310)	(10/307)	(10/307)	(10/307)	(10/307)	(10/307)
Original	55	58	59	58	54	60	55	57	66	55
After 1 hr @0°F	79	90	91	91	85	85	81	82	84	75
Abrasion Index										
Original	112	114			77		102	150	246	71
After 70 hr/212°F	127	132								
Cutgrowth, 50,000 flexes										
Original			150							
After 70 hr/212°F	0						500	500	400	0

* See Appendix A for identification of materials listed by trade name

TABLE III (Cont'd) CHLOROSULFONATED POLYETHYLENE-CONTAINING COMPOUNDS: EXPLORATORY COMPOUNDING

Materials	63-CS-12	63-CS-13	63-CS-14	63-CS-C-2	63-CS-C-3	63-CS-C-4	63-CS-15	63-CS-16	63-CS-17
	a. Recipes (Cont'd)								
Hypalon 40	100	100	100	50	70	70	100	100	100
Neoprene WRT				50	30	30			
XLC Magnesia	5	5	4	5	4	3	5	5	5
PER 200	5	5						5	
HIS11 233	50	35	30	40	30	30	30	50	50
Mistron vapor		15	20		20	20	20		
SRF Black	5	5	5	5	5	5	5	5	5
Butyl oleate	25						25	25	25
TP-90-B		25		20					
DOS	25	25	25		25	10	25		
Stearic acid	2	2		0.5				2	2
Carbowax 4000	2	2			2	2		2	
ZnO				3		3			
Neozone A			1	1	1	1			
Flexzone 3C			2	2	2	2			
Sunolite				1					
Light process oil			25		25	20			
NBC						2			
Litharge				5			2		
Sundex 53							5		
Tetrone A								25	25
Altax							2		2
Methyl tuads	2	2	0.5	0.5	0.5	0.5	1	2	1
Sulfur	1	1	1	1	1	1		1	
Diethylthiourea			1.5	1.5	1.5	1.5			0.5
DOTG			0.5	0.5	0.5	0.5			
	b. Properties at indicated (cure time, min/temp, °F) (Cont'd)								
	(12/310)	(12/310)	(10/307)	(10/307)	(10/307)	(10/310)	(10/310)	(10/310)	(10/310)
Hardness, Shore A									
Original	72	67	60	68	60	70	60	75	75
After 1 hr @ 0°F	90	83	80	85	80	92	80	90	92
Abrasion Index									
Original			151	66	94	74	79		111
After 70 hr @212°F									
Cutgrowth, 50,000 flexes									
Original			500	200	100	500			
After 70 hr @212°F									

TABLE IV. POLYCHLOROPRENE-CONTAINING COMPOUNDS: EXPLORATORY COMPOUNDING

Materials*	(Recipes and Properties)												
	63-C-1	63-CB-1	63-C-2	63-C-CS-1	a. Recipes		63-C-3	63-C-4	63-CB-2	63-CB-3	63-CB-4	63-C-6	63-C-7
					100	100							
Neoprene WRT	100	70							70	80	90	100	100
Budene 501		30							30	20	10		
Necprene W			100	60									
Hypalon 40				40									
ZnO	5	5			5	5				1	1	5	5
Stearic acid	0.5	0.5			0.5	0.5			0.5	0.5	0.5	0.5	
HiSil 233	50	50			50	50			50	25	25	40	35
SRF Black	3	3			5	5			5	3	3	5	5
Mistron vapor			55	55					15	25	25		25
Light process oil					20	20			20				
Thermoflex A									1			1	
XLC Magnesia					0.5	0.5			4	4	4	4	0.5
Neozone D												1	1
TP-90-B	10	10	5	5						15	15	20	10
Butyl oleate			5	5									
DIDA						20							
Neozone A	2	2			1	1			2	1	1		
Sunolite									1	1	1		
Flexzone 3C					2	2			2	2	2		2
Thiuram M					0.5	0.5							0.5
Litharge			5	5									
Tetrone A			2	2									
NA-33	0.5	0.5							0.5	0.5	0.5	0.5	1.5
Diethylthiourea					1.25	1.25							
Altax			1	1									
DOTG					0.5	0.5							
DFG	0.5	0.5							0.5	0.5	0.5	0.5	0.5
Thionex	1	1							1	1	1	1.5	
Sulfur	1	1			1	1						1	1
b. Properties at indicated (cure time, min/temp °F)													
	(12/307)	(12/307)	(12/307)	(12/307)	(10/287)	(10/287)	(10/300)	(10/300)	(10/307)	(10/290)			
Hardness, Shore A													
Original	70	75	55	68	62	65	74	60	55	68			
After 1 hr @ 0°F	80	80	82	90	80	80	83	65	59	78			
Abrasion, Original	72	108	56	51	89	100	76	45	54	48			
After 70 hr @ 212°F													
Cutgrowth, Original	50	500	0	0	0	0	250	0	0	300			
After 70 hr @ 212°F													

* See Appendix A for identification of materials listed by trade name

TABLE IV (Cont'd) POLYCHLOROPRENE-CONTAINING COMPOUNDS: EXPLORATORY COMPOUNDING

Materials	63-C-18	63-C-19	63-C-20	63-C-21	63-C-22	63-C-23	63-C-24	63-C-25	63-C-26	63-C-27	63-C-28
	a. Recipes (Cont'd)										
Neoprene WRT	100	100	100	100	100	100	100	100	100	100	100
XLC Magnesia	1	1	1		1	1	1	1	1	1	1
ZnO	5	5	5	5	5	5	5	5	5	5	5
Neozone A	1	1									
Flexzone 3C	2	2			2			2			
Akroflex C									2	2	2
UOP 88			2	2							
Sunolite	1										
Thermoflex A					2						
Octamine						2					
Flexzone 6-H											2
HiS11 233	45	45	45	45	45	45	45	45	50	50	45
SRF Black	5	5	5	5	5	5	5	5	5	5	5
Stearic acid											
Light process oil	10	15	15		15	15			20	20	15
Circosol 551											
Sundex 53				15							
Diethylthiourea	1.5	1.5	1.75	1.75	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Methyl tuads	0.5	0.5	0.75	0.75	0.5	0.5	0.5	0.5	0.5	0.5	0.5
DOTG	0.5	0.5	0.75	0.75	0.5	0.5	0.5	0.5	0.5	0.5	0.5
NBC					2	2	2	2	2	2	
Sulfur	1	1	1	1	1	1	1	1	1	1	1
	b. Properties at 12 min cure time/307°F (Cont'd)										
Hardness, Shore A											
Original	67	65	66	72	67	67	66	67	69	69	64
After 1 hr @ 0°F	87	75			77	77	78	77	80	81	75
Abrasion Index											
Original	113	98			58	59	56		51	56	61
After 70 hr@212°F											
Cutgrowth											
Original	50										
After 70 hr@212°F		150	200	300	300	500	200	200	600	300	300

TABLE IV (Cont'd) POLYCHLOROPRENE-CONTAINING COMPOUNDS: EXPLORATORY COMPOUNDING

Materials	63-C-29	63-C-30	63-C-31	63-C-32	63-C-34	63-C-37	63-C-38	63-C-39	63-C-40	63-C-41	63-C-42
	a. Recipes (Cont'd)										
Neoprene WRT	100	80	100	100	100	100	100	100	100	100	100
Paracril Ozo		20									
XLC Magnesia	1	1	4	4	2	1	1	1	1	1	1
ZnO	5	5	5	5	5	5	5	5	5	5	5
Akroflex C	2	2	2	2	2						
Flexzone 6-H	2	2	2	2	2						
Flexzone 3C											
Thermoflex A						2	2	2	2		2
NBC											
Octamine											
Stearic acid		1									
HSil 233	45	45	45	45	45	1	1	1	1	1	1
SRF Black	5	5	5	5	5	45	45	45	45	45	45
Light process oil	15					5	5	5	5	5	5
Circosol 591			15	15	15	15	15	15	15		
Butyl oleate											
Wingstay 100						15	15	15	15	15	15
Aminox						2	2	2	2	2	2
Neozone A											
Diethylthiourea	1.5	1.5				1.75	1.75	1.5	1.5	1.5	1.5
DOTG	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Methyl tuads	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Thionex			0.5	0.5							
MA-33			0.5	0.5							
Permalux											
Sulfur	1	1	1	1	1	1	1	1	1	1	1
Litharge					0.5						

	b. Properties at indicated (cure time, min/temp, °F) (Cont'd)										
	(10/307)	(12/307)	(10/307)	(10/307)	(10/307)	(10/307)	(10/307)	(10/307)	(10/307)	(10/307)	(10/307)
Hardness, Shore A.											
Original	64	75	70	75	60	70	65	60	60	62	61
After 1 hr @ 0°F	77	98	80	85	74						
Abrasion Index											
Original	61	58	51	47	53	76	57	73	69	63	64
After 70 hr@212°F											
Cutgrowth, Orig.	300	500	25	25	0	350	25	50	50	0	0
After 70 hr@212°F											

TABLE IV (Cont'd) POLYCHLOROPRENE-CONTAINING COMPOUNDS: EXPLORATORY COMPOUNDING

Materials	63-C-43	63-C-44	63-C-45	63-C-46	63-C-47	63-C-48
a. Recipes (Cont'd)						
Neoprene WRT	100	100	100	90	100	100
Paracril Ozo				10		
ZnO	3	5	5	5	5	5
Stearic acid	1	1	0.5	1	1	1
XLC Magnesia	1.75	1.75	1	1.5	1.75	1.75
HISil 233	45	45	45	45	45	45
SRF Black	5	5	5	5	5	5
Neozone A	2		1			
Thermoflex A						
Wingstay 100						
Aminox						
UOP 88				2	2	2
Flexzone 3C	2	2	2			
Sunproof Jr.				1		
Polyethylene		2			2	2
Light process oil	15	15	15		16	16
TF-90-B				16		
Butyl oleate						
Octamine				1		
Diethylthiourea	1.5	1.5	1.75	1.5	1.5	1.5
Methyl tuads	0.5	0.5	0.5	0.5	0.5	0.5
DOTG	0.5	0.5	0.5	0.5	0.5	0.5
Sulfur	1	1	1	1	1	1
b. Properties at 10 min cure time/307°F (Cont'd)						
Hardness, Shore A						
Original	66	62	69	70	75	69
After 1 hr @ 0°F	75	73		85	83	75
Abrasion Index						
Original	89	109	79	76	63	56
After 70 hr @ 212°F						
Cutgrowth						
Original	300	0	300	600	300	25
After 70 hr @ 212°F						

TABLE V. POLYBLENDED-CONTAINING COMPOUNDS: EXPLORATORY COMPOUNDING
(Recipes and Properties)

Materials*	63- NVB-1	63- NVB-2	63- NVB-3	63- NVB-4	63- NVS-1	63- NVS-2	63- NVB-6	63- NVB-7	63- NVB-8	63- NVB-9	63- NVB-10
Paracril Ozo	90	80	90	80	60	80	80	80	80	80	80
Budene 501	10	20	10	20	20	10	20	20	20	10	20
SBR-1023					20	10					
SBR-1534										10	
Special Low Nitrile Blend											
Turgum S	3	3	3	3	3			3	3	3	3
Octamine	1	1	1	1	1	1	1	1	1	1	
ZnO	3	3	3	3	5	5	3	3	3	3	3
Stearic acid	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5		1.5
Sunolite					1						
HiS11 233	45	45	45	45	40	40	30		30	25	25
Phil Black 0	3	3	3	3	3	3	3	3	3	3	3
TP-90-B	20	20			20	20	20	20	20	25	25
Metalyn 100			20	20							
Sunproof Jr.	1	1	1	1	1	1	1	2	2	1	1
UOP 88	1	1	1	1						2	2
DIDA											
DIOA											
NALCO L-1718								1			
Thermoflex A										1	1
Mistron vapor											
Captax	1.5	1.5	1.5	1.5	1.5	1.5	25	45	15	35	35
Altax	0.5	0.5	0.5	0.5	0.5	0.5	1.5	1.5	1.5	1.5	1.5
Methazate	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
DOTG	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Sulfur (Spider)	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
b. Properties at 10 min cure time/310°F											
Hardness, Shore A,											
Original	60	62	68	65	65	67	68	57	62	55	59
After 1 hr @ 0°F	82	85	95	92	86	100	84	75	78	79	79
Abrasion Index,											
Original	190	218			279	183	111	94	113	90	93
After 70 hr @ 212°F									93		88
Cutgrowth, Original	150	300	0	0	450	500	300	0	0	50	0
After 70 hr @ 212°F								300	50	200	50

* See Appendix A for identification of materials listed by trade name

TABLE V (Cont'd) POLYBLEND-CONTAINING COMPOUNDS: EXPLORATORY COMPOUNDING

Materials	63- NVB-11	63- NVB-12	63- NVB-13	63- NVB-14	63- NVB-15	63- NVB-16	63- NVB-17	63- NVB-18	63- NVB-19	63- NVB-20	63- NVB-21
Paracril Ozo	80				80	80	80	80	80	80	80
Special Low Nitrile Blend											
Batch A		90									
Batch B											
Batch C		90									
Budene 501	10	10	10	10	20	10	10	20	10	20	10
SBR 1504	10					10	10		10		10
Turgum S	3	3	3	3	3	3	3	3	3	3	3
ZnO	3	3	3	3	3	3	3	3	3	3	3
Stearic acid	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Octamine	1	1	1	1	1	1	1	1	1	1	1
Sunproof Jr.	1	1	1	1	1	1	1	1	1	1	1
HiSil 233	30	45	45	45	25	25	30	40	40	40	1
Mistron vapor	35				35	35	30	20	20	20	40
Phil Black O	3	3	3	3	3	3	3	3	3	3	20
Thermoflex A	1				1	1	1	1	1	1	3
DIOA											
TP-90-B	25	10	10	10	5	5	5				
Metalyn 100		10	10	10	20	20	20	25	25	15	
UOP 88	2	2									
Flexzone 3C											
NALCO L-1718											
DOS					2	2	2	2	2	2	2
Captax					1	1	1	1	1	1	1
Altax	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
DOTG	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Methazate	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Sulfur	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
b. Properties at 10 min cure time/310°F (Cont'd)											
Hardness, Shore A											
Original	62	76	78	77	63	63	65	67	66	63	65
After 1 hr @ 0°F	80	100	100	100	80	80	80	82	80	85	84
Abrasion Index, Orig.											
After 70 hr @ 212°F					118	103	98				
Cutgrowth, Orig.	50										
After 70 hr @ 212°F	200				0	25	0	100	20		

TABLE V (Cont'd) POLYBLEND-CONTAINING COMPOUNDS: EXPLORATORY COMPOUNDING

Materials	63- NVB-22	63- NVB-23	63- NVB-24	63- NVB-25	63- NVB-30	63- NVB-31	63- NVB-32	63- NVB-33	63- NVB-34	63- NVB-35	63- NVB-36
	a. Recipes (Cont'd)										
Paracril Ozo	80	80	80	90	80	80	80	80	80	80	80
Budene 501	20	10	20	10	20	20	20	20	20	10	20
SBR 1504		10									
SBR 1023	3	3	3	3	3	3				10	
Turgum S	1	1	1	1	2	2	2	2	2	2	2
Octamine	2	2	2	2						2	2
UOP 88	3	3	3	3	3	3	3	3	3	3	3
ZnO	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Stearic acid	40	40	20	50	20	30	25	25	20	20	45
HISil 233	20	20	30		25	15	20	20	25	25	
Mistron vapor	3	3	3	3	3	3	3	3	3	3	3
Phil Black 0	15	15	15	15	20	20	20	20	10	10	11
TP-90-B											
TCP											
DOA			15	15							
Cumar P25	1	1	1	1			3	3	2	10	11
Sunproof Jr.	1	1	1	1						2	2
NALCO L-1718											
Flexzone 3C	1.5	1.5	1.5	1.5	2	2	2	2			
Captax	0.5	0.5	0.5	0.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Altax	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
DOTG	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Methazate	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Sulfur	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
b. Properties at 10 min cure time/310°F (Cont'd)											
Hardness, Shore A	65	65	60	68	63	56	65	67	65	65	74
Original	90	90	75	88	83	87	98	100	85	85	96
After 1 hr @ 0°F			79	116	98	103	130	125	109	109	200
Abrasion Index, Orig.			83								
After 70 hr @ 212°F	50	0		25	0	0	0	0	0	0	150
Cutgrowth, Original			100								
After 70 hr @ 212°F										95	

TABLE V (Cont'd) POLYBLEND-CONTAINING COMPOUNDS. EXPLORATORY COMPOUNDING

Materials	63- NVB-37	63- NVB-38	63- NVB-39	63- NVB-40	63- NVB-41	63- NVB-42	63- NVB-43	63- NV-44	63- NVB-45	63- NVB-52	63- NVB-53
	a. Recipes (Cont'd)										
Paracril Ozo	80	80	80	80	80	80	80			85	85
Budene 501	20	20	20	20	20	20	20		20	15	15
Chemivic 100								100	80		
ZnO	3	3	3	3	3	3	3	3	3	3	3
Stearic acid	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Octamine	2	2	2	2	2	?	2	1	1	2	2
UVP 88	2	2	2	2	2		2	2	2	2	2
HiSil 233	25	30	30	45	25	25	25	10	25	40	35
Mistron vapor	20	15	15	20	20		20	20	20	25	30
Phil Black 0	3	3	3	3	3	3	3	3	3	3	3
TP-90-B	10	10	11	13	10	10	10	10	10	15	15
DOA	10	10	11	13	10	10	10	10	10	15	15
Cumar P25	2	2	2	2			2	2	2	2	2
Paragon clay						20					
Turgum S					3	3					
UOP 688					2	2		1	1		
Sunproof Jr.								10	10		
Durez 12687											
Captax	1.5	1.5	1.5	1.5	1.5	1.5	1.5			1.5	1.5
Altax	0.5	0.5	0.5	0.5	0.5	0.5	0.5			0.5	0.5
DOTC	0.5	0.5	0.5	0.5	0.5	0.5	0.5			0.5	0.5
Methazate	0.5	0.5	0.5	0.5	0.5	0.5	0.5		0.	0.5	0.5
Amox #1								1	1		
Unads								0.5	0.5		
Sulfur	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
b. Properties at 10 min cure time/310°F (Cont'd)											
Hardness, Shore A	66	70	64	67	63	61	63	76	70	68	65
Original											
After 1 hr @ 0°F	85	90	84	98	86	85	87	96	90	87	82
Abrasion Index, Orig.	186	207	149	250	131	131	134	98	185	137	132
After 70 hr @ 212°F	164	194	141					117	209		
Cutgrowth, Original	0	0	50	150	0	25	0	100	100	0	0
After 70 hr @ 212°F	100	200	250		200	300	150	450	500	150	100

TABLE VI. DEVELOPMENT OF POLYCHLOROPRENE-CONTAINING COMPOUNDS
(Recipes and Test Results)

<u>Ingredients*</u>	<u>63-C-19</u>	<u>63-C-45</u>
	<u>(Parts by weight)</u>	
a. <u>Compounding Recipe</u>		
Neoprene WRT	100	100
XLC Magnesia	1	1
ZnO	5	5
Neozone A	1	1
Flexzone 3C	2	2
HiSil 233	45	45
SRF Black	5	5
Stearic acid		0.5
Light process oil	15	15
Diethylthiourea	1.5	1.75
Methyl tuads	0.5	0.5
DOTG	0.5	0.5
Sulfur	1	1

b. Test Results at 12 min cure time/307°F

Physical properties

Hardness, Shore A		
Original	66	68
After 1 hr @ 0°F	76	77
After 70 hr @ 212°F	75	79
Abrasion Index		
Original	85	76
After 70 hr @ 212°F	93	82
Cutgrowth, %, after 50,000 flexes		
Original	50	50
After 70 hr @ 212°F	300	300

Properties of specimens taken from
soles of Direct Molded Boots:

Hardness, Shore A	65
Aged 70 hr @ 212°F	75
Cutgrowth, %, after 50,000 flexes	
After 70 hr @ 212°F	200
Abrasion Index	
Original	79
Aged 70 hr @ 212°F	83
Bond strength, lb.	190

* See Appendix A for identification of materials listed by trade name.

TABLE VII. DEVELOPMENT COMPOUNDING OF POLYBLEND-CONTAINING RUBBER
(Recipes and Test Results)

<u>Ingredients*</u>	<u>Standard Compound</u>	<u>63-NV-37</u>	<u>63-NV-38</u>	<u>63-NV-39</u>	<u>63-NV-39(x)</u>
a. Compounding Recipes					
Paracril Ozo	100	80	95	90	90
Budene 501		20	5	10	10
Turgum S	3	3	3	3	3
Octamine	1	1	1	1	1
UOP 88					1
NALCO L-1718		1	1	1	
HiSil 233	45	45	45	45	45
Phil Black O	3	3	3	3	3
ZnO	3	3	3	3	3
Stearic acid	1.5	1.5	1.5	1.5	1.5
Sunproof Jr.	1	1	1	1	1.5
Captax	1.5	1	1	1	0.5
Altax	0.5	1	1	1	0.5
DOTG	0.5	0.25	0.25	0.25	0.5
Methazate	0.5	0.25	0.25	0.25	0.5
Sulfur, spider	1.5	1.5	1.5	1.5	1.5
Metallyn 100	20	20	20	20	20
b. Test Results at 10 min cure time/310°F					
<u>Physical Properties</u>					
<u>Hardness, Shore A</u>					
Original	68	65	65	65	66
After 70 hr @ 212°F	78	76	75	76	77
<u>Hardness, Shore A</u>					
After 1 hr @ 0°F	100	96	100	98	98
<u>Abrasion Index</u>					
Original	101	182	122	166	140
After 70 hr @ 212°F	118	180	131	168	147
<u>Cutgrowth after 50,000 flexes, %</u>					
After 70 hr @ 212°F	0	275	0	120	0
<u>Volume Swell in Ref Fuel B, of ASTM, 70/30 Isooctane/Toluene</u>					
After 46 hr, %	18	49	29	35	36
<u>Properties of Specimens taken from soles of Direct Molded Boots</u>					
<u>Hardness, Shore A</u>					
Unaged	66				65
Aged 70 hr @ 212°F	77				75
<u>Abrasion Index</u>					
Unaged	97				121
Aged 70 hr @ 212°F	107				141
<u>Cutgrowth after 50,000 flexes, %</u>					
Aged 70 hr @ 212°F	0				0

* See Appendix A for identification of materials listed by trade name.

TABLE VIII. DEVELOPMENT COMPOUNDING OF STYRENE-BUTADIENE-CONTAINING RUBBER/EPDM RUBBER IN SBR COMPOUNDS
(Recipes and Test Results)

Ingredients*	a. Recipes									
	64- SBR-22-A	64- SBR-23-A	64- SBR-22-B	64- SBR-23-B	64- SBR-22-C	64- SBR-23-C	64- SBR-22-E	64- SBR-23-E	65- SBR-3	65- SBR-8
SBR 1500	100	90	100	90	100	90	100	90		90
SBR 1502									90	
SBR 1504		20		20		20		20	20	20
Royalene X-400	1	1	1	1	1	1	1	1	1	1
Agerite resin D	1	1	1	1	1	1	1	1	1	1
ZnO	1	1	1	1	1	1	1	1	1	1
UOP 88			3	3						
Kastozone 33					3	3				
Flexzone 3C							3	3	3	3
HiSil 233	45	45	45	45	45	45	45	45	45	45
Phil Black O										
Light process oil	10		10		10		10			
Altax	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
DOTG	0.75	0.75	0.75	0.75	0.75	0.75	0.75	0.75	0.75	0.75
Tetrona A	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Sulfur	3	3	3	3	3	3	3	3	3	3
b. Test results at 15 min cure time/310°F										
Ozone Cracking Test at 50 ± 10 pphm & at 100°F for 7 days exposure										
Cracked(C), no crack(NC) After	C 8 hr	C 2 da	C 1 da	NC 7 da	C 1 da	NC 7 da	C 1 da	NC 7 da	C 3 da	C 18 hr
Hardness, Shore A										
Original	63	66	63	65	64	64	65	68	63	66
Aged 70 hr @ 212°F					No Test Made					
Abrasion Index										
Original	140	146	126	129	123	127	123	129	161	
Aged 70 hr @ 212°F			137	135		138		133		
Cutgrowth after 50,000 flexes, % - Original										
Aged 24 hr @ 212°F	175	175		100	No Test Made	240	200	200	150	200

* See Appendix A for identification of materials listed by trade name.

TABLE IX

DEVELOPMENT COMPOUNDING OF STYRENE-BUTADIENE-CONTAINING RUBBER -
EFFECT OF ZnO LOADING IN SBR 1502
(Recipes and Test Results)

<u>Ingredients*</u>	<u>65- SBR-0</u>	<u>65- SBR-3</u>	<u>65- SBR-5</u>	<u>65- SBR-1-S</u>
<u>a. Recipes</u>				
SBR 1502	90	90	90	90
Royalene X-400	20	20	20	20
ZnO		3	5	1
Stearic acid				1
Agerite resin D	1	1	1	1
UOP 88	3	3	3	3
HiSil 233	45	45	45	45
Phil Black O	3	3	3	3
Altax	1.5	1.5	1.5	1.5
DOTG	0.75	0.75	0.75	0.75
Tetrone A	0.5	0.5	0.5	0.5
Sulfur	3	3	3	3
<u>b. Test results at 15 min cure time/310°F</u>				
Tensile strength, psi	2400	1250	1400	2350
Ultimate elongation, %	590	300	320	610
Stress @ 300%	750	1200	1200	650
Hardness, Shore A				
Original	67	70	71	68
Cutgrowth after 50,000 flexes, %				
Aged 70 hr @ 212°F	600	900	900	400
Abrasion Index				
Original	145	83	79	137

* See Appendix A for identification of materials listed by trade name.

TABLE X. DEVELOPMENT COMPOUNDING OF STYRENE-BUTADIENE-CONTAINING RUBBER

Ingredients*	SBR-29	SBR-29b	SBR-76	SBR-78	SBR-93	SBR-110
a. <u>Compounding Recipes</u>						
SBR 1502	90	90	90	90	90	90
Royalene X-400	20	20	20	20	20	20
HiSil 233	50	50	50	50	50	50
KPC Black	3	3	3	3	3	3
Carbowax 4000						1.75
Stearic acid	1	1	1	1	1	1
ZnO					1	2
Sundex 53	10	10			12	10
Cumar MH-1	10					
UOP 88	2	1.0	1.5	1.5	1.0	1
Flexzone 3C	2	1.5	1.5	1.5	1.5	1.5
TP-110			5	5		
Agerite resin D	1	1	1	1	1	1
Captax	1.5	1.5		1.5	1.5	
Altax			1.5			1.5
DOTG	0.5	0.5	0.8	0.8	0.5	0.75
Tetrone A			0.8			
Thionex					0.25	
Methazate	0.5	1.0		0.8	1	1.0
Sulfur	2.0	2.5	2.5	2.5	2.5	2.0
Reogen						

b. Properties at indicated (cure time, min/temp, °F)

	(12/307)	(10/307)	(12/310)	(12/310)	(10/310)	(10/310)
Hardness, Shore A						
Original	57	63	60	60	61	61
After 2 hr @ 0°F	70	72	73	71	73	72
After 70 hr @ 212°F	67	72	70	70	70	68
Abrasion Index						
Original	34	99	107	102	69	118
After 70 hr @ 212°F	140	141	121		87	119
Cutgrowth after 50,000 flexes, %						
After 70 hr @ 212°F	150	150	4	200	250	200
Mooney Scorch, Large Rotor, @ 300°F						
Min. viscosity	78	84	74	76	74	
Scorch time, T ₅ , Min.	5.0	3.8	5.1	5.6	2.5	
Cure time, T ₃₅ , Min.	6.2	5.3	7.5	6.0	3.0	
Ozone Cracking Test @ 50 ± 10 pphm @ 100°F						
No sign of crack after 7 days	7 days	7 days	7 days	7 days	7 days	No data available

* See Appendix A for identification of materials listed by trade name.

TABLE XI

COMPARISON OF FORMULATION OF STANDARD AND SBR, DMS SOLING COMPOUND

<u>Ingredients*</u>	<u>Std. DMS</u> (Parts by weight)	<u>SBR-29b</u>
Polyblend (Paracril Ozo)	100	
Styrene-Butadiene rubber 1502		90
Royalene X-400, EPDM		20
Turgum S	3	
Octamine	1	
Agerite resin D		1
Flexzone 3C		1.5
UOP 88		1.0
HiSil 233	45	50
EPC Black	3	3
ZnO	3	
Stearic acid	1.5	1
Metalyn 100	20	
Sundex 53		10
Sunproof Jr.	1	
Captax	1.5	1.5
Altax	0.5	
DOTG	0.5	0.5
Methazate	0.5	1.0
Sulfur, spider	1.5	2.5
Press cure, Time, Min/Temp, °F	10/310	10/307

* See Appendix A for identification of materials listed by trade name.

TABLE XII. COMPARISON OF PHYSICAL PROPERTIES OF
RUBBER SOLING COMPOUND SBR-29b AND STANDARD DMS

<u>Samples and Footwear</u>		
	<u>Std. DMS</u>	<u>SBR-29b</u>
a. <u>Properties Tested on Press Cure Samples</u>		
Tensile strength, psi	1800	1850
Ultimate elongation, %	680	700
Stress @ 300% elongation, psi	800	480
Hardness, Shore A		
Unaged	70	65
Aged 70 hr @ 212°F	80	74
After 1 hr @ 0°F	100	74
Abrasion Index		
Unaged	102	99
Aged 70 hr @ 212°F	124	141
Vol. Swell after 46 hrs in ASTM Ref. Fuel B at room temperature		
Percent change	18	200
Ozone Cracking Test in Ozone Chamber at 50 ± 10 pphm, ozone		
After 7 days @ 100°F	No sign of crack	No sign of crack
Mooney Viscosity - Large Rotor @ 300°F		
Minimum viscosity	46	84
Scorch time, Min, T ₅	3.0	3.8
Cure time, Min, T ₃₅	3.8	5.3
Measured Specific Gravity	1.23	1.12
Cutgrowth, %, after 50,000 flexes		
Aged 70 hr @ 212°F	0	150
b. <u>Properties Tested on Finished Footwear</u>		
Bond strength, lb		
Rubber outsole to leather	225	155
Rubber soling		
Hardness, Shore A		
Unaged	70	63
Aged 70 hr @ 212°F	80	72
Abrasion Index		
Unaged	99	93
Aged 70 hr @ 212°F	110	131
Cutgrowth, %, after 50,000 flexes		
Aged 70 hr @ 212°F	25	55
Ozone Cracking Test in Ozone Chamber w/50 ± 10 pphm ozone		
7 days @ 100°F	No sign of crack	No sign of crack

TABLE XIII

VULCANIZING CEMENT FOR THE STANDARD DMS COMPOUND

Masterbatch

<u>Ingredients</u>	<u>Parts by Weight</u>
Paracril CLT	100
Octamine	1.5
ZnO	10
Phil Black O	40
Stearic acid	1
Cumar P25	15
Durez 12687	40
Total	207.5

	<u>Part A</u>	<u>Part B</u>
Masterbatch	207.5	207.5
Captax	5	
Methazate	1	
DOTG	1	
Spider sulfur		5.0
Total	214.5	212.5
MEK	645	639

Immediately before use, parts A and B are equally mixed. (See Appendix A)

TABLE XIV

VULCANIZING CEMENT FOR SBR RUBBER SOLE COMPOUND

Masterbatch

<u>Ingredients</u>	<u>Parts by Weight</u>
SBR 1502	100
Agerite Resin D	1
EPC Black	45
ZnO	5
Stearic acid	<u>1.5</u>
Total	152.5

	<u>Part A</u>	<u>Part B</u>
Masterbatch	152.5	152.5
Sulfur	6	
Captax		3
Methazate		<u>2</u>
Total	<u>158.5</u>	<u>157.5</u>

TABLE XV

WELCO RO-SEARCH MODIFIED SBR FORMULATIONS

Soling and Cement Compound

<u>a. Soling Compound</u>		<u>b. Vulcanizing Cement</u>	
<u>Ingredients*</u>	<u>Parts by Wt.</u>	<u>Ingredients*</u>	<u>Parts by Wt.</u>
SBR 1502	84	Neoprene AC	100
Royalene X-400, EPDM	20	MgO	4
ZnO	2	ZnO	5
HiSil 210	50	HiSil 233	25
Philliprene 1603	9	Agerite stalite	2
Agerite resin D	1	Durez 12687	40
Stearic acid	1		
Flexzone 3C	1.5	<u>Add to solvent</u>	
UOP 88	1.5	A-1 Thiocarbanilide	4
Sundex 790	7		
Captax	0.82	<u>Solvent:</u>	
Methazate	0.25	2.25 parts toluene	
Carbowax 4000	1.75	2.25 parts MEK	
Sulfur, spider	2.0	1.00 part neoprene 620	

* See Appendix A for identification of materials listed by trade name.

TABLE XVI

TEST DATA - WELLCO RO-SEARCH FABRICATED DMS BOOTS USING SBR COMPOUND

<u>Properties Tested</u>	<u>Molded Slabs</u>	<u>Samples from Finished Boots</u>
Hardness, Shore A		
Original	63	63
After 1 hr @ 0°F	73	
After 70 hr @ 212°F	70	71
Abrasion Index		
Original	92	88
Aged 70 hr @ 212°F	93	89
Cutgrowth, %, after 50,000 flexes		
Aged 70 hr @ 212°F	100	0
Ozone resistance @ 50 \pm 10 pphm ozone and @ 100°F		
After 7 days exposure	No crack	No crack
Original, stress-strain		NA
Tensile strength, psi	2400	
Ultimate elongation, %	840	
Stress @ 300% elongation, psi	300	
Bond strength, lb		
Original	NA	170
After 7 days @ 158°F	NA	140
Bond strength, dead weight application of 20 pounds		
After 4 hr @ room temperature	NA	No appreciable separation
After 4 hr @ 158°F	NA	No appreciable separation

APPENDIX A

COMPOUNDING MATERIALS

Trade Name, Identification and Supplier (Code)*

<u>Materials (Trade Name)</u>	<u>Identification</u>	<u>Supplier*</u>
Agerite Powder	Phenyl-B-Naphthylamine	18
Agerite Resin D	Polymerized trimethyl dehydro-quinoline	18
Akroflex C	35% N N'Diphenyl-P-phenylendiamine	4
Altax	Benzothiazyl disulfide	4
Amax #1	N-oxydiethylone-benzothiazole-2-sulfenamide	18
Aminox	Reaction product of diphenylamine and actone	10
Budene 501	Cispolybutadiene type rubber	19
Butyl eight	Activated dithiocarbamate	18
Butyl oleate		20
Butyl stearate		20
Carbon black, Types		
EPC	Easy processing channel	2,7
MPC	Medium processing channel	2,7
Phil Black 0	High abrasive furnace	12
SRF	Semi-reinforcing furnace	2,7
Captax	2-Mercaptobenzothiazole	18
Carbowax 4000	Polyethylene glycol	21

* See Appendix B for name of supplier

APPENDIX A (Cont'd)

COMPOUNDING MATERIALS

<u>Materials (Trade Name)</u>	<u>Identification</u>	<u>Supplier</u>
Chemivic 100	Butadiene-Acrylonitrile/ Polyvinylchloride blend	19
Circosol 551	Naphthenic type oil	15
Circosol 591	Naphthenic type oil	15
Cumar MH-1	Coumarene-idene resin medium grade	11,1
Cumar P25	Para coumarene-indene resin	1
DBP	Dibutyl phthalate	8,22
DIDA	Di-isodecyl adipate	8
Diethylene glycol		20
Diethylthiourea		4
DIOA	Di-isooctyl adipate	8
DOA	Di-aryl adipate	21
DOS	Di-aryl sebacate	21
DOTG	Di-orthotolylguanidine	4
DPG	Di-phenylguanidine	8
Durez 12687	Phenolic resin	23
Dutrex 20	Aromatic Hydrocarbon oil, (no longer supplied)	
Eastozone 33	N,N'-bis(1,4 dimethylphenyl-p- phenylenediamine	22
Flexol TOF	Tri (2-ethylhexyl) phosphate	21
Flexzone 3C	N-isopropyl-N'-phenyl-p- phenylenediamine	10

APPENDIX A (Cont'd)

COMPOUNDING MATERIALS

<u>Materials (Trade Name)</u>	<u>Identification</u>	<u>Supplier</u>
Flexzone 6-H	N-phenyl-N'-cyclohexyl-p-phenylene diamine	10
HiSil 233	Hydrated silica	13
Hycar 1072	Carboxylic acid terpolymer	5
Hypalon 40	Chlorosulfonated polyethylene	4
Light process oil	Naphthenic oil (light colored)	25
Litharge	Lead oxide	24
XLC magnesia	Extra light magnesium oxide	25
Metalyn 100	Methylated tall oil ester	6
Methazate	Zinc dimethyl dithiocarbamate	10
Methyl tuads	Tetramethylthiuram disulfide	4
Mistron vapor	Magnesium silicate	14
NA-22	2-Mercaptiomidazoline	4
NA-33	Modified mercaptiomidazoline (no longer supplied)	
NALCO L-1718	Polyalkanalpolyamine	9
NBC	Nickel butyl carbamate	4
Neoprene W	Polychloroprene	4
Neoprene WRT	Polychloroprene	4
Neozone A	N-phenyl-alpha-naphthylamine	4
Neozone D	N-phenyl-beta-naphthylamine	4
Octamine	A reaction product of diphenylamine & diisobutylene	10

APPENDIX A (Cont'd)

COMPOUNDING MATERIALS

<u>Materials (Trade Name)</u>	<u>Identification</u>	<u>Supplier</u>
Paracril CLT	Butadiene-acrylonitrile copolymer	10
Paracril Ozo	Nitrile-polyvinylchloride resin blend	10
Paragon clay	Hydrated aluminum silicate	7
PER 200	Pentacrythritol	6
Permalux	Di-ortho-tolylguanidine, salt of dicatechol borate	4
Philliprene 1603	Styrene-butadiene rubber, master- batched with carbon black	12
Red iron oxide		28
Reogen	Mixture of an oil soluble sulfonic acid w/high molecular weight	18
Royalene X-400	Ethylene-propylene diene-oil extended rubber	10
Santocure	N-cyclohexyl-2-benzothiazole sulfenamide	8
SBR, type	Styrene-butadiene copolymer	
1500		10, 12, 29
1502		10, 12, 29
1504		10, 12, 29
1023		10, 12, 29
1778		19
1712c		19
Stearic acid		20
Sulfur		18

APPENDIX A (Cont'd)
COMPOUNDING MATERIALS

<u>Materials (Trade Name)</u>	<u>Identification</u>	<u>Supplier</u>
Sundex 53	Aromatic type oil	15
Sundex 790	Aromatic type oil	15
Sunolite wax	Selected blend of waxy hydrocarbons	26
Sunproof Jr.	Mixture of selected waxes	10
TCP	Tricresylphosphate	8,13
Tetrone A	Dipentamethylene thiuram tetrasulfide	4
Thermoflex A	p-p dimethoxydiphenylamine and 25% diphenyl-p-phenylene diamine	4
Thionex	Tetramethylthiuram monosulfide	4
Thiuram M	Tetramethylthiuram disulfide	4
Triethanolamine		20
TP-90-B, plasticizer	A high molecular weight polyether	16
TP-110, plasticizer	Polyether plasticizer	16
Turgum S	Terpene resin acid blend	7
Unads	Tetramethylthiuram monosulfide	18
UOP 88	N,N'-bis(1-methyl heptyl)-p- phenylene diamine	17
UOP 688	Unsymetrically substituted phenylene diamine	17
Wingstay 100	Mixture of diaryl-p-phenylene diamine	19
ZnO	Zinc oxide	20,27

APPENDIX B
MATERIAL SUPPLIERS

Number

1. Allied Chemical Corporation, Philadelphia, Pennsylvania
2. Cabot Corporation, Boston, Mass.
3. Diamond Alkali Company, Cleveland, Ohio
4. E. I. duPont deNemours and Company, Inc., Wilmington, Delaware
5. B. F. Goodrich Chemical Company, Cleveland, Ohio
6. Hercules Powder Company, Wilmington, Delaware
7. J. M. Huber Corporation, New York, N. Y.
8. Monsanto Chemical Company, Akron, Ohio
9. Nalco Chemical Company, Chicago, Illinois
10. Naugatuck Chemical Company, Naugatuck, Connecticut
11. Neville Chemical Company, Pittsburgh, Pennsylvania
12. Phillips Petroleum Co., Rubber Chemicals Div., Akron, Ohio
13. Pittsburgh Plate Glass Company, Pittsburgh, Pennsylvania
14. Sierra Talc and Clay Company, So. Pasadena, California
15. Sun Oil Company, Philadelphia, Pennsylvania
16. Thiokol Chemical Corporation, Trenton, New Jersey
17. Universal Oil Products, Des Plaines, Illinois
18. R. T. Vanderbilt Company, Inc., New York, N. Y.
19. Goodyear Chemicals, Akron, Ohio
20. The C. P. Hall Company, Akron, Ohio
21. Union Carbide Corporation, New York, N. Y.
22. Eastman Chemical Products, Kingsport, Tennessee

APPENDIX B (Cont'd)

MATERIAL SUPPLIERS

Number

- 23. Hooker Chemical Corp., Durez Plastics Div., Niagara Falls, N.Y.
- 24. National Lead Company, New York, N. Y.
- 25. Marine Magnesium Products Div., Merch Co. Inc., Rahway, N. J.
- 26. Witco Chemical Company, Inc., New York, N. Y.
- 27. St. Joseph Lead Company, New York, N. Y.
- 28. Eaton Chemical & Dye Stuff Company, Detroit, Michigan
- 29. Goodrich Gulf Chemicals, Cleveland, Ohio

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13. ABSTRACT A new direct molded sole rubber compound designed primarily to give better traction at low temperature, and on snow and ice, than the present standard vinyl-modified Buna N rubber compound, has been successfully developed by utilizing Styrene-Butadiene rubber (SBR), type 1502. The vulcanizates of this new compound exhibited good physical properties which are comparable with the physical properties exhibited by the present standard sole compound. In addition, this new compound also displayed excellent ozone resistance and good low-temperature behavior - a property which makes this compound more suitable for wear on snow and ice. Furthermore, the test results on the finished boot samples showed that the new SBR compound has satisfied not only the curing conditions imposed by the CEMA and Welco process but also the physical requirements, except for solvent resistance, which are specified in Military Specification, MIL-B-43154, for the standard direct molded sole (DMS) combat boot.		

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Development	8					
SRR Rubbers	8,9		9			
Rubber	9					
Synthetic rubber	9					
Vulcanizates	9					
Low-temperature	0					
Direct-molded sole (DMS)	4		9			
Combat footwear	4		9			
Testing			8			
Evaluation			8			
Physical properties			9			
Traction			9			
Wear resistance			9			
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